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L10: Entry 191 of 229 File: USPT Apr 21, 1981

DOCUMENT-IDENTIFIER: US 4263011 A
TITLE: Crystallization process

clm 23/300

DEPR:
The bed of solid particles utilized in the process of this invention can be agitated to keep the particles in continuous motion by any suitable means. For example, agitation can be provided by mechanical vibration, by pulsations induced by a pump, by the use of ultrasonics, by rotating impellers, by rotating stirring devices equipped with flexible or rigid blades, by Archimedean-screw stirrers, and so forth. In using rotating stirring devices, these will typically be rotated at a speed in the range from about 50 revolutions per minute to about 2000 revolutions per minute. The agitation should be such as to ensure homogeneous conditions concerning temperature and concentration at all points in the medium wherein crystallization occurs.

DEPR:
In utilizing the process of this invention for the continuous manufacture of radiation-sensitive silver halide emulsions, the silver salt and the halide salt or salts are separately introduced into the reaction chamber. The reaction medium is typically an aqueous medium, and water soluble silver salts, such as silver nitrate, and water soluble halide salts, such as alkali halide salts, most commonly sodium or potassium halide salts, are introduced into the reaction chamber. In an aqueous medium the peptizer is typically a hydrophilic colloid, such as gelatin, which can be introduced with either or both of the silver and halide salts or separately therefrom. It is recognized that the silver and the halide salts can include any conventional counter ion which allows for desired solubility in the reaction medium and which is not incompatible with the silver halide grain-forming reaction. A wide variety of both aqueous and nonaqueous silver halide grain-forming double-jet reaction techniques and reactants for use therein are taught in the art. A thorough discussion of conventional silver halide precipitation reactions can be found in the following references: Photographic Chemistry, Pierre Glafkides, Fountain Press, London, 1958, pp. 327-330; Nucleation in Silver Bromide Precipitation, C. R. Berry and D. C. Skillman, J. Phys. Chem., 68, 1138-43 (1946); C. R. Berry and D. C. Skillman, J. Photo Sci., 68, 121-133 (1964); and The Theory of the Photographic Process, Third Edition, C. E. K. Mees and T. H. James, Macmillan, 1966, Chapter 2; Berry, Rate Processes in AgBr Crystal Growth, Photog. Sci. and Engr., 18, 4-8, (1974); Berry, A New Model for Double-Jet Precipitations, Photog, Sci. and Engr., 20, 1-4, (1976).

DEPR:
This example illustrates the use of the method of this invention in a reaction-crystallization process in which silver bromide crystals are formed by reaction of potassium bromide and silver nitrate.

DEPR:
An apparatus similar to that illustrated in FIG. 1 having a cylindrical glass chamber 1, which is 4 centimeters in diameter and 10 centimeters in height, was partially filled with glass beads 5 having a diameter of 2 millimeters. Chamber 1 was filled to level NN with a one molar solution of potassium nitrate at 40.degree. C. and agitator 2 was started and operated at a speed of 1000 revolutions per minute. By means of volumetric pumps, a one molar solution of potassium bromide containing 3% of acylated gelatin was introduced through inlet tube 6 at a flow rate of 5 milliliters per minute and a one molar solution of silver nitrate was introduced through inlet tube 7 at a flow rate of 5 milliliters per minute. The potassium bromide and silver nitrate solutions were each at a temperature of 40.degree. C. and chamber 1 was insulated against heat

L10: Entry 215 of 229 File: USPT Sep 21, 1976

DOCUMENT-IDENTIFIER: US 3981973 A
TITLE: Crystal growth in the presence of finely divided polytetrafluoroethylene

BSPR:
Crystals are rarely pure because they generally contain small quantities of foreign matter which have been built-in or occluded. Gases, liquids and solids are readily occluded in a growing crystal; dirt, air and mother liquor are the most common occlusions found in commercial crystals. From the commercial crystallization point of view, the main interest lies in finding methods to prevent such occlusions from occurring. Vapor occlusions are minimized by avoiding vigorous agitation or boiling. The application of ultrasonic radiation to the system is also used to prevent bubbles or particles from adhering to a growing crystal face. Most importantly, the crystallizing system is kept clean to avoid dirt, other debris and particularly organic contaminants from being occluded into a crystal.

BSPR:
While it is clearly advantageous to produce crystals as large as possible, their actual size is only of secondary importance; what really matters, with relatively large crystals, is the regularity of the product. The less regular the crystals, the fewer voids there will be between crystals. Moreover, with less regular, relatively small crystals, crystalline fines are generally present which given rise to objectionable dust which makes handling the crystals in bulk a most unpleasant task. Of course, besides being essentially dust-free, uniform, large crystal masses have many other desirable properties; they can be filtered and washed more efficiently during processing, they have good flow characteristics, and they have a pleasing appearance--an important sales factor, particularly in the sale of big amounts of ammonium nitrate, ammonium sulfate, and nickel sulfate. Thus, there is a need for a simple, economical method of producing relatively large, well-defined crystals which may be grown from a supersaturated solution, and which are essentially free from dusty crystalline fines.

423/445; 23/200
23/302 R